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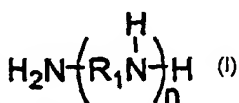
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- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(54) Title: AQUEOUS POLYAMINOAMIDE AND SURFACTANT COMPOSITION FOR PAPERMAKING



dispersion in papermaking.

(57) Abstract: This invention is directed to an aqueous dispersion comprising one or more polyaminoamides, one or more surfactants and water, wherein the dispersion has a pH of from about 5 to about 8 and the polyaminoamides are prepared by reacting one or more fatty acids with one or more polyamines of formula (I) wherein R<sub>1</sub> is C<sub>2</sub>-C<sub>4</sub> alkylene and n is 2, 3, 4 or 5 and use of the

AQUEOUS POLYAMINOAMIDE AND SURFACTANT COMPOSITION FOR PAPERMAKING  
TECHNICAL FIELD

This invention concerns compositions for use in papermaking and methods of using the compositions in a papermaking process to produce paper having enhanced optical and dimensional properties.

BACKGROUND OF THE INVENTION

The process of paper manufacture is a dynamic process in which a wide range of additives are introduced to a slurry of pulp to produce a sheet with desired physical properties. Each additive is selected to impart a desired property to the paper sheet, resulting from the additive's own physical properties, or the additive's interaction with other slurry components to produce a desired property. For example, the sheet optical properties of brightness (reflection of incidental light from sheet surface) and opacity (refraction and/or diffusion of incidental light) and are important features which may determine the market value of the sheet.

The use of inorganic pigments and/or fillers is well known in the art. Specifically, titanium dioxide powder is sometimes used by the papermaker to achieve sheet optical properties. Although titanium dioxide provides the useful property enhancing sheet opacity and brightness, its use is often limited by its high cost and difficulty in handling, introduction, and retention into the pulp slurry.

Additionally, kaolin clay has also been used as alternate pigment/filler in papermaking to improve the sheet properties. Calcined kaolin clay is typically suspended in an aqueous dispersion via continuous

agitation prior to introduction into the pulp slurry. Without appropriate dispersion of the kaolin particles using both mechanical and chemical dispersion stabilization techniques, the particles are subject to quick agglomeration and sedimentation resulting in non-uniform paper sheet properties and sheet defects.

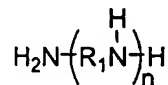
Further, the aforementioned inorganic pigments and fillers are difficult to retain in the forming paper sheet. Large density gradients between the pulp fibers and the inorganic minerals such as titanium dioxide and clays often lead to gravitational migration of these minerals out of the forming web during the dewatering process of the forming paper sheet. Non-retained minerals travel out of the sheet during dewatering, sometimes depositing in the paper machine wire fabric, causing significant paper machine downtime. Charged polymers commonly known as coagulants and/or flocculants may be added to improve retention of these minerals in the forming sheet, but such practice increases the complexity and expense of the papermaking process. More ideally, the composition imparting the intended sheet property should be self-retaining in the forming sheet. Further, the ideal composition would be of a density more similar to that of the suspended fiber slurry thereby reducing the effect of selective migration of the sheet property functional additive during the sheet dewatering process and resulting in more uniform paper sheet property profile across the axis normal to that of the papermachine direction.

Methods of enhancing the brightness, opacity and sizing using a composition comprising the mono- and di-fatty acid amide of aminoethylethanolamine and a surfactant are disclosed in U.S. Patent Nos. 5, 292,363, 5,296,024 and 5,393,334. These compositions, although

effective, are not as efficient as the aqueous dispersions described herein.

#### SUMMARY OF THE INVENTION

In its principal aspect, this invention is directed to an aqueous dispersion comprising one or more polyaminoamides, one or more surfactants and water, wherein the dispersion has a pH of from about 5 to about 8 and the polyaminoamides are prepared by reacting one or more fatty acids with one or more polyamines of formula



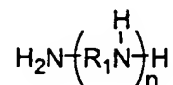
wherein  $\text{R}_1$  is  $\text{C}_2\text{-C}_4$  alkylene and  $n$  is 2, 3, 4 or 5.

Adding the aqueous dispersion of this invention to the pulp slurry during papermaking results in a uniform dispersion of the composition in said paper and improved sheet opacity and brightness. There is minimal regression of sheet mechanical properties such as sheet tensile and tear strength, and this invention requires a lower dosage than current organic sheet enhancing additives, for example the composition disclosed in U.S. Patent No. 5,296,024.

Accordingly, in another aspect, this invention is directed to a method of increasing opacity, bulk or brightness in a paper sheet comprising

- a) adding to a pulp slurry of cellulose fibers an aqueous dispersion comprising one or more polyaminoamides, one or more surfactants and water, wherein the dispersion has a pH of from about 5 to about

8 and the polyaminoamides are prepared by reacting one or more fatty acids with one or more polyamines of formula



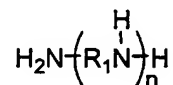
wherein  $\text{R}_1$  is  $\text{C}_2\text{-C}_4$  alkylene and  $n$  is 2, 3, 4 or 5; and

b) forming the slurry into the paper sheet.

#### DETAILED DESCRIPTION OF THE INVENTION

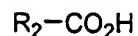
##### Definitions of Terms

"Polyamine means a compound of formula



wherein  $\text{R}_1$  is divalent straight or branched chain saturated hydrocarbon radical of from two to about four carbon atoms and  $n$  is 2, 3, 4 or 5. Preferred polyamines are those in which  $\text{R}_1$  is ethylene, i.e. diethylenetriamine, triethylenetetramine, tetraethylenepentamine and pentaethylenehexamine. Triethylenetetramine is more preferred.

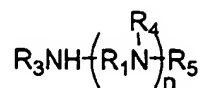
"Fatty acid" means a compound of formula



wherein  $\text{R}_2$  is the hydrocarbon side chain of a saturated or unsaturated fatty acid. Preferably,  $\text{R}_2$  is a straight chain hydrocarbon of from about 13 to about 22 carbon atoms.  $\text{R}_2$  may contain one or more double bonds. Preferred fatty acids include behenic acid, stearic acid,

myristic acid and oleic acid. Stearic acid is more preferred.

"Polyaminoamide" means a compound of formula



wherein  $R_3$ ,  $R_4$  and  $R_5$  are independently selected at each occurrence from H and  $R_2C(O)-$  and  $n$  is 2, 3, 4 or 5, provided that at least one of  $R_3$ ,  $R_4$  and  $R_5$  are H and at least one of  $R_3$ ,  $R_4$  and  $R_5$  are  $R_2C(O)-$ . In a more preferred aspect, at least two of  $R_3$ ,  $R_4$  and  $R_5$  are H and at least one of  $R_3$ ,  $R_4$  and  $R_5$  are  $R_2C(O)-$ .

"Brightness" means the numerical value of the blue-light reflectance of white, near-white and naturally colored pulp, paper and paperboard. The higher the blue-light reflectance, generally the whiter the products will appear. Standard methods and apparatus for measuring brightness are described in the official Tappi test method T 452 om-98.

"Opacity" means the extent to which a single sheet of white or near-white paper hides printed matter on underlying sheets of similar paper. Standard methods and apparatus for measuring brightness are described in the official Tappi test method T 519 om-91.

"Bulk" means the specific volume of the paper sheet expressed as  $cm^3/g$  and determined using Tappi official test method T 220 om-88.

#### Preferred Embodiments

The polyaminoamide is prepared by reacting the polyamine and fatty acid at high temperature, preferably from about 300 °F to about 350 °F until water evolution ceases. The polyamine starting material may be a single

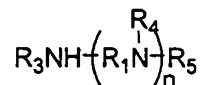
polyamine or a mixture of polyamines. Likewise, the fatty acid may also be a single compound or a mixtures of fatty acids. The resulting polyaminoamide is a hydrophobic cationic amide with a designed molecular structure having superior molecular dimensional properties. The polyaminoamide is typically an amorphous solid or wax.

The aqueous dispersion of this invention is prepared by melting the polyaminoamide, generally at a temperature of from about 200 °F to about 250 °F and then slowly adding one or more surfactants with stirring. The heat is then removed and hot water is added to achieve the desired concentration. The pH of the dispersion is then adjusted to about 5-8, preferably to about 6-7 with a protic acid, preferably acetic acid. The resulting aqueous dispersion contains from about 5 weight percent to about 25 weight percent, preferably from about 10 weight percent to about 20 weight percent polyaminoamide.

The surfactant or surfactants are added to reduce the surface tension of the composition to insure adequate dispersion of the polyaminoamide. Surfactants suitable for preparing the aqueous dispersion of this invention contain polyoxyethylene as the polar group and have a HLB of from about 11 to about 16. If a mixture of surfactants is used, the surfactants are selected such that the mixture has a HLB of from about 11 to about 16. Representative surfactants include nonylphenol and dodecylphenol ethoxiates, tallow amine (TAM) ethoxylates, ethoxylated castor oil, ethoxylated stearates, tallates and oleates, C<sub>9</sub>-C<sub>11</sub> primary alcohol ethoxylates, ethoxylated tridecyl alcohols, ethoxylated primary and secondary aliphatic alcohols, ethoxylated sorbitan alkylates, and the like. Preferred surfactants or

surfactant mixtures have a HLB of about 14. Tallow amine ethoxylate is more preferred.

In a preferred aspect of this invention, the polyaminoamide has formula



wherein  $R_2$  is the hydrocarbon side chain of a saturated or unsaturated fatty acid;  $R_3$ ,  $R_4$  and  $R_5$  are independently selected at each occurrence from H and  $R_2C(O)-$  and  $n$  is 2, 3, 4 or 5, provided that at least one of  $R_3$ ,  $R_4$  and  $R_5$  are H and at least one of  $R_3$ ,  $R_4$  and  $R_5$  are  $R_2C(O)-$ .

In another preferred aspect, at least two of  $R_3$ ,  $R_4$  and  $R_5$  are H and at least one of  $R_3$ ,  $R_4$  and  $R_5$  are  $R_2C(O)-$ .

In another preferred aspect, the polyaminoamide is prepared by reacting two molar equivalents of the fatty acids with one molar equivalent of the polyamines.

In another preferred aspect, the polyamines are selected from diethylenetriamine, triethylenetetramine, tetraethylenepentamine and pentaethylenehexamine.

In another preferred aspect, the fatty acid is selected from behenic acid, stearic acid, myristic acid and oleic acid.

In another preferred aspect, the polyamine is triethylenetetramine and the fatty acid is stearic acid.

In another preferred aspect, the aqueous dispersion has a pH of from about 6 to about 7.

In another preferred aspect, the surfactants contain a polyoxyethylene group as the polar group and have a HLB of from about 11 to about 16.

In another preferred aspect, the surfactants have a HLB of about 14.



In another preferred aspect, the surfactant is tallow amine ethoxylate.

The dimensional properties of the resulting composition exhibit enhanced interfacial properties when added to a pulp slurry during the papermaking process. The resulting paper exhibits the enhanced sheet properties of brightness, opacity, and sheet bulk.

The pulp slurry is a slurry of cellulose fibers derived from chemical, mechanical or chemo-mechanical treatments of substances containing cellulose fibers such as wood. This slurry may also contain recycled paper or paperboard products. The aqueous dispersion is added after the cellulose fibers have been sufficiently treated to make them ready for the papermaking process. For example, after they have been bleached to remove lignins and deinked if recycled paper pulp is being used. In general, the aqueous dispersion will be added to a thick stock (2-4 wt/wt percent) of the pulp slurry before dilution with recycled paper machine water or addition of inorganic fillers.

The aqueous dispersion is added to the pulp slurry in an amount of from about 0.10 to about 10.0 active lbs / ton, preferably from about 0.3 to about 6.0 active lbs./ton. The preferred addition point is to the machine chest, before dilution of the thick stock with whitewater, where there is a contact time with the furnish components of at least about 10 minutes before the system is diluted.

The aqueous dispersion of this invention may be used along with conventional inorganic fillers, dyes and retention aid programs. The dispersion is preferably added to the pulp slurry before the addition of any additional papermaking additives.

In a preferred aspect, the aqueous dispersion is used in conjunction with one or more retention and drainage aids. The selection of the retention and drainage program will depend on the type of furnish being treated. In general each individual furnish is to some extent unique and the type and amount of retention/drainage aid required will be optimized by laboratory testing. For example the mill may be operating in an acid or alkaline condition which will control which inorganic fillers are used. Clay is generally used in acid while calcium carbonate is preferred in alkaline systems. Titanium dioxide will often be added to either type of system.

Representative retention and drainage programs used by those knowledgeable in papermaking may contain cationic coagulants such as the reaction product of epichlorohydrin with dimethylamine with or without added ammonia, ammonia - dichloroethane condensates, poly(ethyleneimine), poly(diallyldimethylammonium chloride) or other suitably prepared cationic products. Inorganic coagulants such as alum or poly(aluminum chloride) may also be used separately or in conjunction with the above organic cationic coagulants. Frequently, cationic starch is also added to the furnish and will act as a cationic coagulant.

Cationic flocculants which may be used are usually selected from copolymers of acrylamide with dimethylaminoethylacrylate or dimethylaminoethyl methacrylate which have been quaternized with methylchloride. Also used are acrylamide copolymers with diallyldimethylammonium chloride.

Anionic flocculants which may be used can be selected from copolymers of acrylamide with acrylic acid

(sodium salt) and acrylamidomethylpropane sulfonic acid (sodium salt).

The retention and drainage program may also use an anionic colloidal particle such as silica, bentonite or cross-linked acrylamide - acrylic acid polymers.

The foregoing may be better understood by the following Examples, which are presented for illustration and are not intended to limit the scope of the invention.

#### Example 1

A representative polyaminoamide is prepared as follows:

Stearic acid (79.17 grams, Industrene B - an industrial grade which is a mixture of stearic and palmitic acids) is mixed with 20.83 grams of triethylenetetramine (TETA) and heated to 300 ° F. A slow nitrogen sparge is initiated with mixing and the reaction mixture is heated to 350 ° F and held for four hours. The reaction mixture is then cooled. The product is the distearamide of triethylenetetramine as determined by NMR.

#### Example 2

A representative aqueous dispersion according to this invention is prepared as follows:

The distearamide of Example 1 (110 g) is heated to 230 - 250 ° F and a tallow amine ethoxylate dispersing agent (TAM-15, 5 g,) is added with mixing. The heat is removed and 880 grams of hot water are added slowly with mixing. After completion of the water addition, cooling is initiated and 5 grams of acetic acid are added to adjust the pH to 6 - 7. The resulting aqueous dispersion contains 11 wt % of the triethylenetetramine distrearamide.

### Example 3

The effectiveness of the aqueous dispersions of this invention is determined using a standard acid furnish prepared with and without added clay (kaolin #2 middle Georgia high brightness). Handsheets are prepared (using a British Sheet Mold) at varied levels of the aqueous dispersion and compared to handsheets prepared without the aqueous dispersion.

The fiber source for the sheets is a mixture of 80/20 wt/wt % hardwood/softwood kraft which is repulped from dry lap and beaten to a freeness of 350 - 370 CSF. This fiber furnish is then diluted to a concentration of 0.5 wt % with tap water and adjusted to pH 5.0 with dilute acetic acid. A clay slurry, at 0.5 wt % concentration, is also prepared. Handsheets are then made with a grammage of 65 grams/sq. meter containing varied levels of the organic modifier and clay. This is done by removing a given volume of the 0.5% fiber and substituting with the same volume of 0.5% clay.

The aqueous dispersion of this invention is added to the appropriate furnish with stirring 15 minutes before sheet formation and a retention program of alum, cationic starch and a cationic flocculant is added 1 minute prior to sheet formation. The handsheets are pressed and dried under standard Tappi conditions, T 205 om-88. Routinely, 7 sheets are made at each condition and averages taken of the optical and mechanical properties. The optical properties were measured on an Elrepho instrument using Tappi standard T519 to determine opacity. The results are shown in Table 1.

Table 1

## Effect of TETA Distreamide on Sheet Opacity

Clay Level %	Opacity		
	0 lb/ton	2 lb/ton	4 lb/ton
0	76.7	77.0	77.3
5	78.8	78.8	79.1
10	80.9	81.2	81.5

As shown in Table 1, in each case whether with or without clay addition to the sheet there is measurable increase in the opacity of the handsheet produced. The laboratory data also show that the addition of 1 lb of clay would result in an opacity increase of 0.02065 units. Therefore, the TETA distreamide can be used to replace 15 to 30 lbs. of clay.

In a similar manner, a furnish from a newsprint machine containing 14.5% calcium carbonate ash is treated with the TETA distreamide composition of Example 2 at various dosages and handsheets are made. The brightness is determined as shown in Table 2.

Table 2

## Effect of TETA Distreamide on Sheet Brightness

Brightness			
0 lb/ton	2 lb/ton	4 lb/ton	8 lb/ton
49.03	49.54	49.65	50.33

## Example 3

The aqueous dispersion of this invention is compared to the composition of US Patent 5,292,363 as follows.

In a furnish containing 8.4 wt/wt % clay as described in Example 2 both the TETA distearamide of this invention and the stearamide of aminoethylaminoethanol (US Pat. 5,292,363) are used to treat the furnish and opacities are measured. In order to obtain the same opacity increase produced by 1.45 lbs of the aqueous dispersion of this invention, 3.33 lbs were required of the composition of US Patent 5,292,363.

#### Example 4

In a separate experiment the aqueous dispersion of TETA distearamide of this invention is used to treat an 80/20 HW/SWK furnish containing no filler. Rather than air drying (Tappi standard T -205 om-88) the handsheets produced, the sheets are dried on a drum dryer at 215 °F and then measured for opacity. The results are shown in Table X below .

Table 3

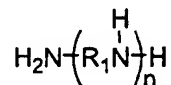
Effect of TETA Distearamide on Heat-Dried Sheet Opacity

Clay Level %	Opacity		
	0 lb/ton	2lb/ton	4 lb/ton
0	75.1	78	80.89

In this case, heated drying appears to have a much greater effect on the increase in sheet opacity than that involved in air drying as shown in Table 1.

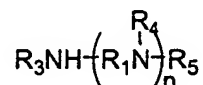
CLAIMS

1. An aqueous dispersion comprising one or more polyaminoamides, one or more surfactants and water, wherein the dispersion has a pH of from about 5 to about 8 and the polyaminoamides are prepared by reacting one or more fatty acids with one or more polyamines of formula



wherein  $\text{R}_1$  is  $\text{C}_2$ - $\text{C}_4$  alkylene and  $n$  is 2, 3, 4 or 5.

2. The aqueous dispersion of claim 1 wherein the polyaminoamide has formula



wherein  $\text{R}_2$  is the hydrocarbon side chain of a saturated or unsaturated fatty acid;  $\text{R}_3$ ,  $\text{R}_4$  and  $\text{R}_5$  are independently selected at each occurrence from H and  $\text{R}_2\text{C}(\text{O})-$  and  $n$  is 2, 3, 4 or 5, provided that at least one of  $\text{R}_3$ ,  $\text{R}_4$  and  $\text{R}_5$  are H and at least one of  $\text{R}_3$ ,  $\text{R}_4$  and  $\text{R}_5$  are  $\text{R}_2\text{C}(\text{O})-$ .

3. The aqueous dispersion of claim 2 wherein at least two of  $\text{R}_3$ ,  $\text{R}_4$  and  $\text{R}_5$  are H and at least one of  $\text{R}_3$ ,  $\text{R}_4$  and  $\text{R}_5$  are  $\text{R}_2\text{C}(\text{O})-$ .

4. The aqueous dispersion of claim 1 wherein the polyaminoamide is prepared by reacting two molar equivalents of the fatty acids with one molar equivalent of the polyamines.

5. The aqueous dispersion of claim 1 wherein the polyamines are selected from diethylenetriamine, triethylenetetramine, tetraethylenepentamine and pentaethylenehexamine.

6. The aqueous dispersion of claim 1 wherein the fatty acid is selected from behenic acid, stearic acid, myristic acid and oleic acid.

7. The aqueous dispersion of claim 1 wherein the polyamine is triethylenetetramine and the fatty acid is stearic acid.

8. The aqueous dispersion of claim 1 that has a pH of from about 6 to about 7.

9. The aqueous dispersion of claim 1 wherein the surfactants contain a polyoxyethylene group as the polar group and have a HLB of from about 11 to about 16.

10. The aqueous dispersion of claim 7 wherein the surfactants have a HLB of about 14.

11. The aqueous dispersion of claim 8 wherein the surfactant is tallow amine ethoxylate.

12. A method of increasing opacity, bulk or brightness in a paper sheet comprising adding the aqueous dispersion of claim 1 to a pulp slurry of cellulose fibers and forming the slurry into the paper sheet.



## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US01/40012

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(7) :D21H 17/07, 17/14

US CL :162/158

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 162/158, 179

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2,772,969 A (REYNOLDS et al) 04 DECEMBER 1956, see entire document	1-12
Y	US 5,393,334 A (HUTCHESON) 28 February 1995, see entire document.	1-12



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
*A* document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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Date of mailing of the international search report

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